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The Investigation and Production of Titanium-Tantalum Junctions
Diffusion Bonded at High Temperature (855°C to 920°C): the
Influence of Temperature, Time Pressure and Roughness on the
Mechanical Properties, and the Optimisation of the Bonded
Conditions

by

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THE INVESTIGATION AND PRODUCTION OF TITANIUM-TANTALUM
JUNCTIONS DIFFUSION BONDED AT HIGH TEMPERATURE (855 to 920°C):
THE INFLUENCE OF TEMPERATURE, TIME, PRESSURE AND ROUGHNESS ON
THE MECHANICAL PROPERTIES, AND THE OPTIMISATION OF THE BONDED CONDITIONS

ÉTUDE ET RÉALISATION DE JONCTIONS TITANE-TANTALE
SOUDÉES PAR DIFFUSION À HAUTE TEMPÉRATURE (855 - 920°C):
INFLUENCE DES PARAMÈTRES TEMPÉRATURE, TEMPS, PRESSION
ET RUGOSITÉ SUR LES PROPRIÉTÉS MÉCANIQUES ET
OPTIMISATION DES CONDITIONS DE SOUDAGE

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AUTHOR'S SUMMARY

This investigation shows that, despite a clear Kirkendall effect, it is possible within the technical constraints to find a combination of the four main operating parameters (temperature, time, pressure and roughness) allowing the successful diffusion bonding of titanium and tantalum (with fracture of the tensile test specimens occurring in the tantalum at 360 MPa). The optimum temperature range at low pressure (2 to 5 MPa) lies in the α phase of titanium at 875 to 885°C. The pressure, which plays a decisive role in the resorption kinetics of residual defects, must be greater than 2 MPa to ensure the production of strong and ductile junctions in a reasonable time. The application of a temporary overpressure during heating offers many advantages, such as limited deformation of test pieces, saving of time, the production of better quality junctions, and even the bonding of surfaces with significant roughness.

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1 INTRODUCITON

Users of tantalum (the chemical industry for its resistance to corrosion, the aeronautical industry for its toughness, also the nuclear and armament industries) seek to achieve economies in the amount of this very expensive material employed. This may be done by attaching components in less expensive materials such as titanium to components in tantalum by means of diffusion bonding, *eg* for reinforcements in containers, etc.

The choice of titanium for this kind of assembly is determined by several criteria; titanium, like tantalum, possesses a good corrosion resistance, which is important for the applications envisaged; it has a better specific mechanical strength and the tantalum-titanium equilibrium diagram (Fig 1¹) is simple, without intermetallic compounds. Details of the diffusion coefficients are provided in the literature. The coefficient of heterodiffusion \tilde{D} of tantalum in β titanium at infinite dilution and 1000°C is $5.2 \times 10^{-10} \text{ cm}^2 \text{ s}^{-1}$ (Ref 2). Fedotov *et al*³ have determined by the method of Matan the change in the coefficient of mutual diffusion of tantalum and titanium as a function of the titanium content of Ti-Ta alloy bonded to a block of pure tantalum at 1000°C ($D \approx 6 \times 10^{-11} \text{ cm}^2 \text{ s}^{-1}$ at 10 at .% tantalum). These data show that diffusion bonding of tantalum and titanium is achievable at this temperature, and probably at lower temperatures in the region of 900°C.

The choice of the technique of assembly by diffusion bonding is justified by:

- the difficulties encountered in the application of the more standard techniques (TIG or EB welding), in particular regarding the significant differences in the fusion temperatures of the materials to be bonded.
- the advantages of the method which, by permitting the production of solid-state bonding at moderate temperature, allows:
- the control of any structural transformations which may occur by a judicious alteration of the bonding parameters;
- the bonding of thin items without risk of penetration;
- the limitation of the deformation of test pieces, especially if the operation is carried out in an isostatic press;
- the production of assemblies having a very narrow diffusion zone, thus eliminating the problem of contamination into the tantalum.

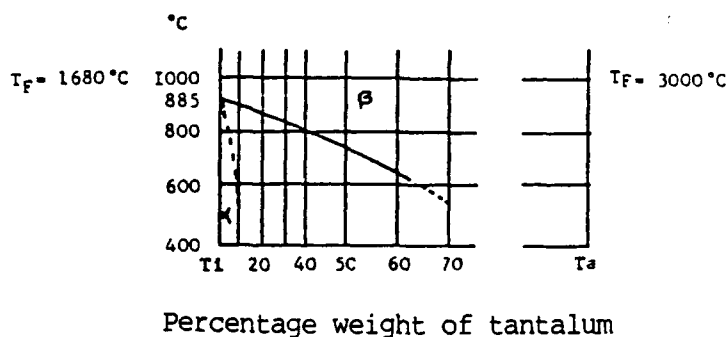


Fig 1 Titanium-tantalum diagram (after Hansen¹)

In simple terms, diffusion bonding is carried out in two principal stages⁴⁻⁶.

- The first stage consists of the establishment of physical contact between the two surfaces by flattening the rough surface features, and depends principally on the condition of the surface, the pressure and the temperature. It commences during heating and continues to the start of the maintained temperature condition.
- The second stage is that of diffusion, during which the residual defects resulting from superficial roughness must be resorbed and a solid metallic bond established. This stage mainly depends, of course, on temperature and time, but also on the outcome of the first stage.

The object of this investigation was to produce junctions stronger than tantalum, which is the weaker material. We therefore went on to examine the influence of the principal operating parameters: temperature, bonding period, applied pressure and the roughness of the surfaces to be joined. We had earlier investigated the phenomena involved in straightforward diffusion of a tantalum-titanium couple.

2 EXPERIMENTAL TECHNIQUES

2.1 Material and samples employed

For this investigation we used pure tantalum (having 50 ppm total impurities), body cubic centred up to the melting temperature, and with a breaking strength of 250 MPa at 20°C, also commercially pure titanium (Table 1 below for analysis of principal impurities) having a breaking strength of 477 MPa. The temperatures of the start and finish of the $\alpha_{\text{hcp}} \rightarrow \alpha_{\text{hcp}} + \beta_{\text{bcc}}$ transformation were determined by dilatometry and by investigation of the grain size; the β nucleation is accompanied by a refining of the α grains following return to ambient temperature, while transition to the β phases induces an

exaggerated growth of the grain and modification to its morphology. The start of the $\alpha \rightarrow (\alpha + \beta)$ transformation occurs between 895 and 905°C; the end of the $(\alpha + \beta) \rightarrow \beta$ transformation occurs between 910 and 920°C.

The samples were made up from tantalum sheet 2 mm thick placed as a 'sandwich' between two titanium parallelepipeds 9 mm thick, the surfaces to be bonded measuring 8 mm \times 10 mm. The configuration of these samples allowed two junctions to be produced during each test.

Table 1

	C	Si	Fe	N ₂
ppm by weight	120	<250	240	250

As explained below (section 2.3.2), this configuration of the test pieces is particularly severe for tensile tests, and provides more complete information than could be obtained using two identical blocks of titanium and tantalum.

After bonding, a thin sample is taken from the assembly for metallography, and a tensile test piece of diameter 5 mm is machined from what remains.

2.2 Experimental apparatus

The bonding of the samples is carried out under secondary vacuum in a vertical tubular enclosure heated by a sliding tubular heater. A demountable assembly (Fig 2) designed and built by one of us⁷ allows the samples for bonding to be introduced, positioned and held under pressure in the enclosure. The samples, two per experiment, are stacked in the lower part of the assembly. The upper part includes a pneumatic ram which, via sliding and spacing rods, allows the application of a pressure of from 2 to 70 MPa, based on the area of 80 mm² of the samples to be bonded. Two thermocouples placed level with each of the two samples indicate the temperature with an accuracy of $\pm 3^\circ\text{C}$.

2.3 Checks on bonding

2.3.1 Metallographic examination

Final polishing of the metallographic plate of the sandwiched Ti-Ta-Ti assemblies starts with diamond paste of 1 μm particle size, and finishes using a vibrating polisher with a suspension of alumina in a glycerine-alcohol mixture, in order to eliminate residual 'spreading' at the interface. It should be noted

that prolonged polishing with this machine causes fissuring of the residual defects in the junctions.

The usual micrographic etching is not possible, on account of the difference in the solution potentials of the two bonded metals. We therefore used ion bombardment to reveal the structure of the diffusion zone, also the joining of the grains of the different materials.

We formally defined a 'percentage contact' by relating the cumulative lengths of the diffusion zones without defect to the total length of the junction examined. This was carried out on unetched, polished samples by measuring the length of the pores parallel to the plane of the junction:

N_1 = number of pores whose length is less than or equal to 3 μm ,

N_2 = number of pores whose length is between 3 and 6 μm ,

Σl = the sum of the lengths of the pores greater than 6 μm .

The percentage contact is then equal to:

$$\left| 1 - \frac{1.5 N_1 + 4.5 N_2 + \Sigma l}{\text{total length}} \right| \times 100 \quad .$$

This allows us to follow the development of the joint.

We checked the validity of the measurement by comparing results obtained with different samples bonded under identical conditions, or on different parts of the same sample. With the exception of tests carried out at 895°C, in which the allotropic transformation of tantalum disturbed the observations, the measurement is quite reliable (most commonly within $\pm 2\%$, or $\pm 4\%$ in the worst cases).

2.3.2 Tensile tests

The results of tensile tests, which provide information on strength, ductility and fracture location (either at the interface or within the tantalum) give a very good indication of the quality of the junction. However it must be noted that, on account of the thin section of the tantalum, it is not able to undergo plastic deformation when its elastic limit is exceeded, because the titanium is still in its elastic condition; there is a confinement effect. Therefore the strength of the assembly, measured with this configuration of tensile test piece, may exceed the breaking strength of tantalum (250 MPa) and reach 340 to 360 MPa before fracture in the tantalum occurs. It is therefore a

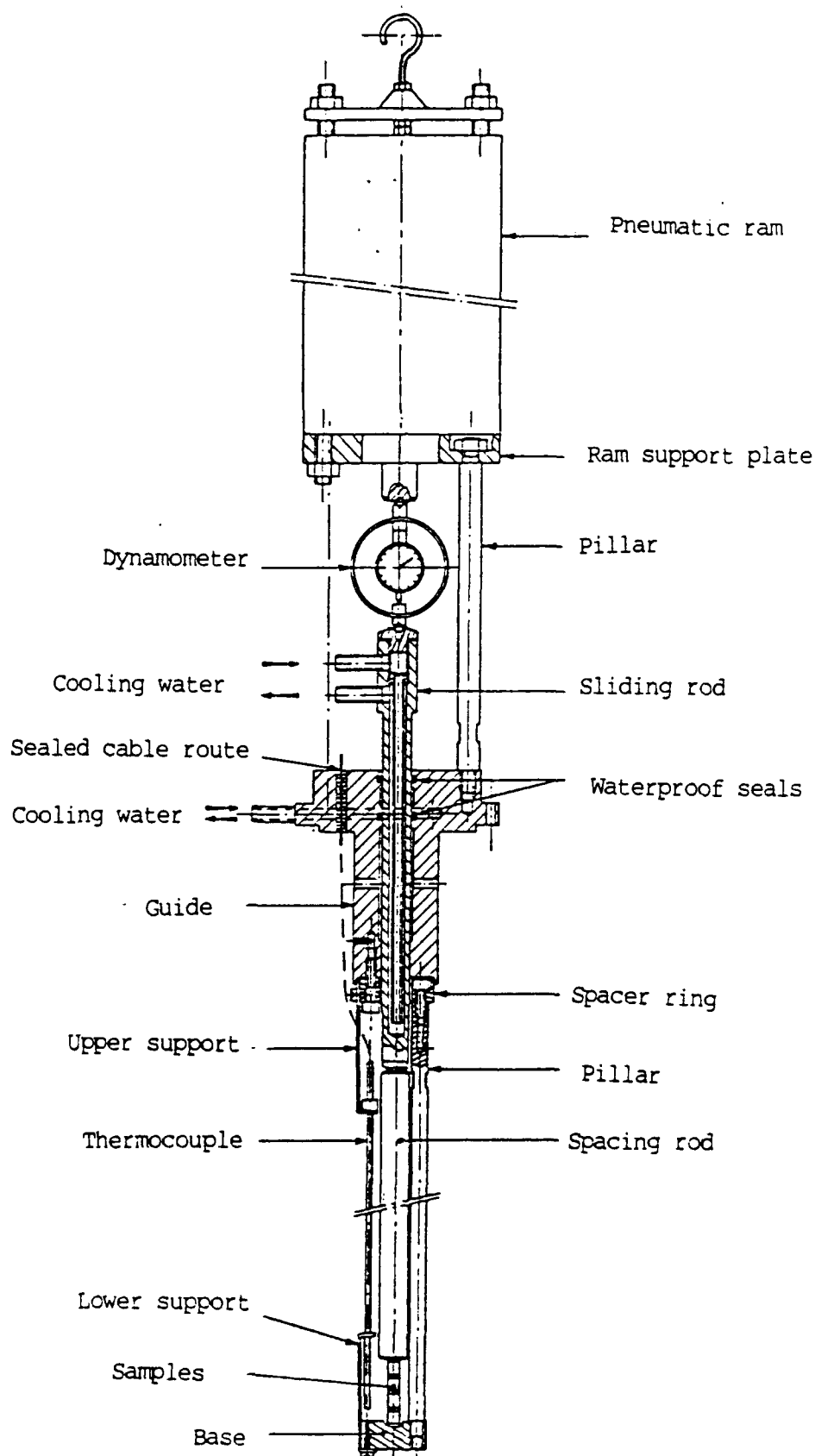


Fig 2 Demountable assembly allowing samples for bonding to be held under pressure

very severe test when compared with one carried out on a sample made up of equal parts of tantalum and titanium. The severity of the test is further accentuated when the tantalum undergoes plastic flow, considering the triaxial constraints which obtain at the interface, and which are significant by reason of the thinness of the tantalum. Thus, assemblies which exhibit fracture in the tantalum provide a good guarantee of integrity.

3 RESULTS AND DISCUSSION

3.1 Investigation of the phenomena of diffusion

Before investigating the optimum bonding conditions, we determined which of the two metals diffused the more rapidly into the other, and considered the significance of the Kirkendall effect. To do this we set up some diffusion processes over a period of 100 h at 880 and 920°C in the α and β phases respectively of the titanium employed.

The chemical nature of the elements making up the diffusion couple which were of interest to us did not allow us to make use of the usual markers such as tungsten. We therefore considered that the residual defects observed in the first samples of bonding - defects which were a consequence of the initial roughness of the surfaces to be bonded (section 3.2.1.1 and Figs 3 and 4) - could act as inert markers.

In order to be able to follow the movement of the interfaces we:

- used a very thin sheet of tantalum (0.31 mm) in a manner which enabled us to observe and measure its change of thickness using a scanning electron microscope at low magnification;
- took a reference sample of the Ti-Ta-Ti couple at the end of 1 h of diffusion from the material treated for 100 h at 880°C, and at the end of 4 h for that treated for 100 h at 920°C. A pressure of 50 MPa, to ensure bonding, was applied during this preliminary period;
- compared the position of the interfaces noted on the reference sample (1 h and 4 h diffusion periods, at 880 and 920°C respectively) with that of the tantalum-diffusion zone interface observed after 100 h of the isothermal condition without the applied pressure.

Examination of the micrographic sections of the Ti-Ta-Ti diffusion couples showed (Table 2 and Fig 3) that:

- the thickness of the sheet of tantalum is very little reduced relative to the extent of the diffusion zone. It is mainly the titanium that is affected by the diffusion.
- the residual defects migrate during the diffusion process. This displacement, which is towards the titanium, is much more significant at 920°C than at 880°C. This confirms that there is a clear Kirkendall effect, that the defects act well as markers and that the titanium is the element which diffuses quickest. It should be noted, furthermore, that we observe in the process carried out at 920°C an enlargement of the defects, supporting the hypothesis that the Kirkendall effect has an influence, and the defects behave like porosity. In the samples subjected to 880°C we observed more of a resorption of defects; the Kirkendall effect is distinctly less pronounced here, than at 920°C.

Table 2

Displacement of interfaces and defects following a 100 h isothermal process

	Process temperature			
	880°C		920°C	
	Process period			
	1 h	100 h	4 h	100 h
Mean thickness of Ta (μm)*	305	297	300	280
Mean depth of diffusion zone (μm)	11	133	38	280
Distance between the centre-lines of the mean position of the defects (μm)*	312	325	310	353

* Measurements from micrographs derived from a scanning electron microscope

- the diffusion zone is largely made up of a two-phase structure formed during cooling. It is bordered on the tantalum side by the solid α solution

which is relatively narrow, and on the titanium side, for those processes only in the α phase of the titanium, by the solid α phase of the titanium, which is also very narrow (Fig 1). Fig 4b is representative, at a reduced depth by reason of the shorter diffusion time, of this general shape of the diffusion layers.

Since the diffusion zone has an essentially two-phase structure, it is not possible to carry out precise measurements of its composition by means of X-ray microanalysis (an EDAX system of analysis fitted with an energy-selective

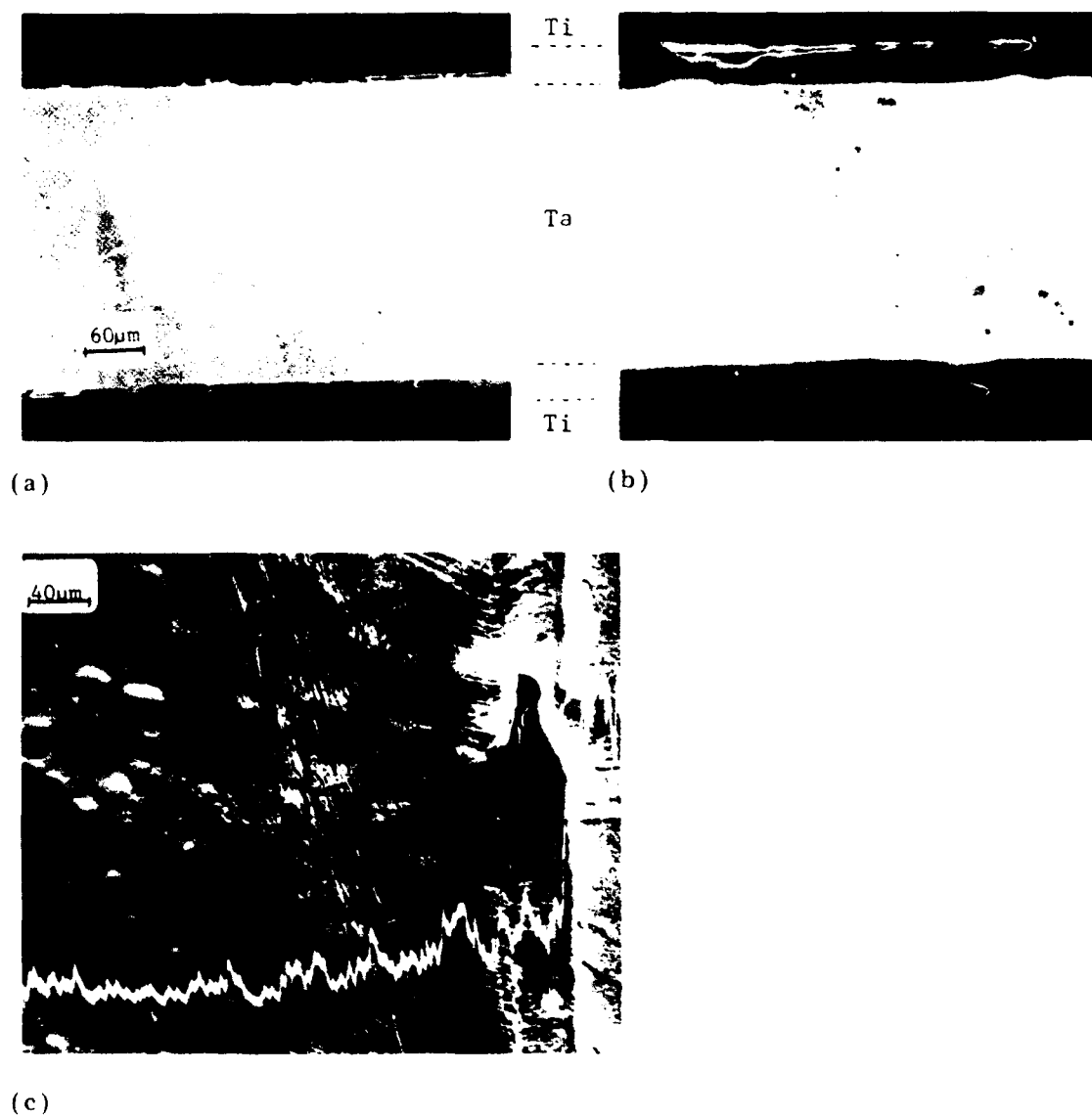


Fig 3 Long-duration diffusion test at 920 C: (a) sampled after 4 h of diffusion; (b) 100 h test; (c) concentration profile of tantalum (line M) in the diffusion zone of the 100 h test.

spectrometer), even more so since the microstructure becomes coarser with distance from the tantalum. (This variation in the degree of fineness of the phases depends on the temperature of the $\beta \rightarrow (\alpha + \beta)$ transformation which varies with composition (Fig 1). As a consequence, we were interested in the single-phase β zone, and we used the EDAX program of ZAF (Z-absorption-fluorescence) corrections without standard. In this program we assumed that the sum of the percentages of the elements making up the sample was equal to 100, which was a justifiable hypothesis in view of the purity of the metals employed and our check on the absence of any pollution related to the isothermal process.

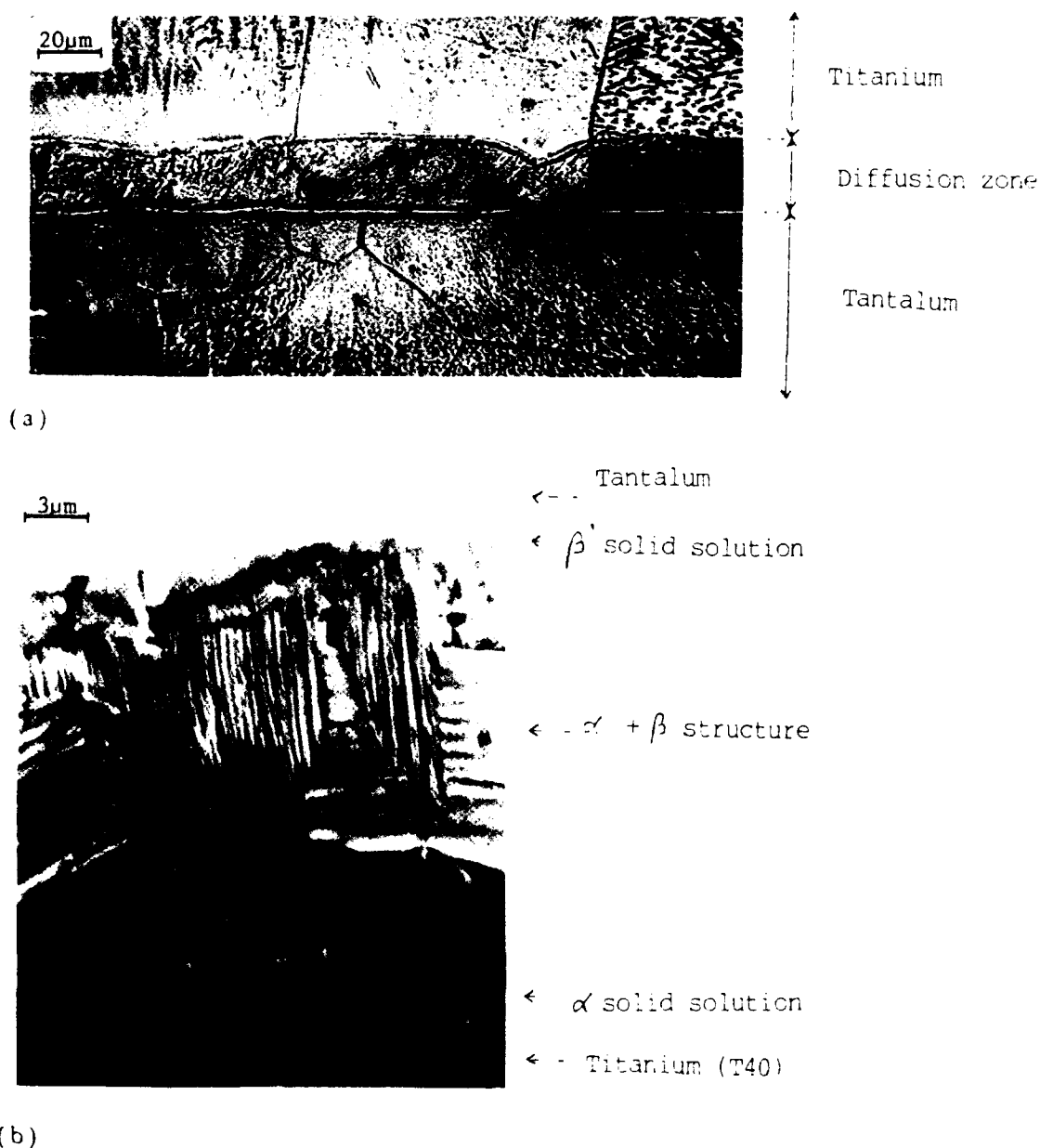


Fig 4 Micrographs of the titanium-tantalum diffusion zone of an assembly subjected to 875 C at 5 MPa, for 4 h: (a) optical microscope; (b) scanning microscope

With regard to the samples processed for 100 h, all the spot checks carried out in the tantalum at a distance greater than one micron from the tantalum-diffusion zone interface provided confirmation that the interface seen through the scanning electron microscope, after polishing, corresponded in practice to an interface of pure tantalum with the zone of diffusion.

The analyses carried out on the sample treated for 100 h at 920°C in the 40 μm single-phase β zone adjacent to the tantalum show a large variation in the composition over a relatively short distance from the diffusion zone (Table 3). Thus at 2 μm from the pure tantalum interface the content of titanium has already reached 30% by weight. The variation in composition is thereafter very slow in the two-phase zone, changing from 61% to 100% over a distance of 240 μm . Fig 3c shows the concentration profile of tantalum (line M) recorded during a linear scan of the diffusion zone. In the sample processed at 880°C the single-phase zone measures only 23 μm , but the concentration profile exhibits the same form.

Table 3

Proportion of titanium in the β layer of the diffusion zone

Distance from the pure Ta interface (μm)	2	5	20	38
% Ti by weight	30	38	53	61

This rapid variation in the composition explains why the boundary in the micrographs is so clearly defined between the pure tantalum and the diffusion zone. In fact, this is consistent with an equally rapid variation in the mechanical properties of these two zones, demonstrated by differential erosion during mechanical polishing. The resulting relief creates a significant contrast between the pure tantalum and the diffusion zone. Moreover, this rapid variation in composition is accompanied by a significant contrast in the distribution of the elements in each zone, adding to the other effect. Hence we obtain the impression in the micrographs of a discontinuity between the pure tantalum and the diffusion zone, particularly in those made with the scanning electron microscope (Figs 3 and 4).

This investigation demonstrates that:

- the titanium diffuses more rapidly than the tantalum.
- there is a clear Kirkendall effect which is particularly significant when the diffusion process is carried out in the β phase of the titanium. In this case it causes an enlargement of the residual defects acting as markers, which may be prejudicial to the mechanical behaviour of the bond. However, the Kirkendall effect does not necessarily appear to be penalising when the isothermal treatment is carried out in the α phase of the titanium.
- the narrow single-phase β zone corresponds to a large variation in composition, in support of the apparent discontinuity observed in the micrographs between the pure tantalum and the diffusion zone.
- the degree of diffusion is sufficient to allow bonding to be achieved in a reasonable time.

3.2 The influence of the different operating parameters on the quality of Ti-Ta-Ti diffusion-bonded junctions

A preliminary investigation⁷ showed that the application of pressure throughout the entire temperature rise favoured the establishment of physical contact between the surfaces to be bonded, and enabled the production of junctions which were better than those obtained without pressure during heating. In this case, the flow of the rough surface features is on the one hand found to be very limited, and on the other, the diffusion initiated in these features causes them to become hardened. This effect thus limits their subsequent flattening on application of pressure at the temperature of the test, causing an increase in the quantity of residual defects and the production of weaker junctions. As a consequence, in all our tests, pressure was applied for the entire period of the temperature rise (30 to 40 minutes).

3.2.1 Influence of the temperature of diffusion bonding

The range of temperature employed (855 to 920°C) covers the α , $\alpha + \beta$, and β phases of titanium⁸; this allowed us to investigate the influence of the allotropic transformation of titanium on the quality of the junctions obtained.

During this investigation, the conditions of pressure, temperature and surface were as follows:

- a constant pressure of 2 MPa;
- periods of 1 h and 6 h at each temperature;

- a surface condition achieved by polishing with No.600 paper, followed by chemical etching in fluoronitric solution. This contributed to a maximum depth of detected roughness, R_{\max} , of $2\text{ }\mu\text{m}$, and an R_a of $0.2\text{ }\mu\text{m}$ (R_a being the arithmetic mean of the absolute values of all the departures of the roughness profile from the mean line).

After each test, the bonded samples were measured in order to check the deformation during bonding. The values given in Table 4 are those for the deformation of a sample of titanium of 9 mm thick (the flow of tantalum over this range of temperature being negligible compared with that of titanium). These results clearly show that the deformation of the test pieces, which is very small at temperatures within the α phase of the titanium, increases substantially when the test is performed in the $(\alpha + \beta)$ phase, and reaches destructive levels at the temperatures of the β phase. This is to be expected from the well-known ductility of titanium in the β phase.

3.2.1.1 Metallographic investigation

The micrographs of the junctions bonded at 875°C and 5 MPa for 4 h (Fig 4a&b) demonstrate that:

- some defects of various shapes and sizes remain: they occur as a result of the initial roughness of the bonded surfaces. Depending on whether they are small or large at the end of the first stage, they are at the end of the diffusion stage either totally enclosed (and distorted) within the diffusion zone or in contact with the tantalum (Fig 4a).
- the diffusion zone does not maintain a constant thickness along the interface (Fig 4a); this is on account of the delay in diffusion occurring in the non-contacting zones (due to insufficient flattening of the rough surface features). In the presence of too great a size of defect, it is even possible to have a discontinuity of the diffusion zone.
- this diffusion zone is largely made up of a needle-like Widmanstätten structure (known as a 'basketwork structure') bounded on the tantalum side by the β solid solution rich in tantalum (Fig 4b). For junctions produced in the α phase of the titanium (at a temperature of less than 895°C), the diffusion zone is bounded on the titanium side by a very narrow band of solid solution (Fig 4a&b) clearly defining the diffusion zone. On the other hand, for junctions produced in the $(\alpha + \beta)$ or β phases, the needles of the Widmanstätten structure merge into the titanium, and the diffusion zone is not clearly defined. This

Table 4

Results of tests of diffusion bonding of the tantalum-titanium couple as a function of bonding temperature

Period of maintained temperature																
1 h										6 h						
Bonding temperature (°C)																
	855	865	885	895	895	905	910	920	855	865	885	895	895	905	910	920
Breaking strength (MPa)	239	282	350	306	266	191	184	97	279	266	357 364	348	224	239	294	320
Extension at fracture (A%)	0	0	7	0	0	0	0	0	0	0	14 10	7	0	0	0	0
Fracture location*	Int	Int	Int	Int	Int	Int	Int	Int	Int	Int	Int Int	Int	Int	Int	Int	Int
Percentage contact (%)	46	61	85	63	64	50	33	50	49	58	82 75	83 54	44	53	69	81
Deformation of Ti sample (mm)	0.05	0.03	0.03	0.07	0.05	0.21	0.14	1.4	0.05	0.03	0.035 0.04	0.17 0.13	0.03	0.22	0.12	1.83
Apparent width of diffusion zone (μm)	10	14	14	21	18	21	21	18	20	26	34 30	42 42	38	44	36	40
Ti grain size (μm)	171	159	181		142	97	92		222	281	206	121	156	113	145	212

Bonding pressure, 2 MPa;
polishing with No.600 paper:

* Int, at the interface

microstructure is in good agreement with that to be expected by the equilibrium diagram (Fig 1).

The 'apparent' width of the diffusion zone (from about 10 to 40 μm according to the tests under consideration), and very easily measurable from the micrographs, doubles in value as the temperature changes from 855 to 920°C, during the steady condition (1 h or 6 h), or when the bonding period is changed from 1 to 6 h, at constant temperature.

The percentage contact of the junctions changes as a function of bonding temperature in quite a surprising manner (Fig 5).

- at temperatures in the α phase (855 to 885°C), the percentage contact increases steadily with temperature; however the values corresponding to periods of 1 h and 6 h are very close, and the bond does not appear to improve with time.
- at temperatures in the $(\alpha + \beta)$ phase (895 to 910°C), the percentage contact is considerably reduced when compared with the values obtained in the α phase, and passes through a minimum. The difference in percentage contact between the 1 h and 6 h tests becomes more marked at the highest temperatures.
- at a temperature of 920°C (β phase of titanium) the percentage contact is clearly increased when compared with that for tests carried out in the $(\alpha + \beta)$ phase, and for both periods under consideration. The difference between the results obtained for the 1 h and 6 h bonding periods becomes more marked.

We attribute these variations to the effects of the structural transformation of titanium*: the nucleation of the β phase (bcc), less dense than the α phase (hcp), may give rise to constraints and local deformations, especially in the still fragile junctions being established during heating. After the transformation point has been passed, these constraints may encourage fracture of the weakest points of bonding, causing a considerable drop in the measured percentage contact. This continues to diminish while the quantity of β phase being formed increases, namely, when the bonding temperature is greater in the $(\alpha + \beta)$ phase of the titanium. However, the ability of the system to repair this damage grows with increase in ductility (improvement in physical contact) and with diffusion (re-bonding). This is why we observe a distinct improvement in

* This effect appears to be negligible in the case of homogeneous titanium-tantalum bonding⁹.

the percentage contact at higher temperatures, and in the β phase in particular. The rise in the curve (corresponding to 1 h duration) with temperature is also explained by this mechanism; a certain amount of damage is already repaired at a high temperature sustained for 1 h. These results show that even when the process is carried out in the β phase of the titanium, the Kirkendall effect is not injurious for the periods under consideration; the applied pressure perhaps also plays a part in opposing the enlargement of the defects.

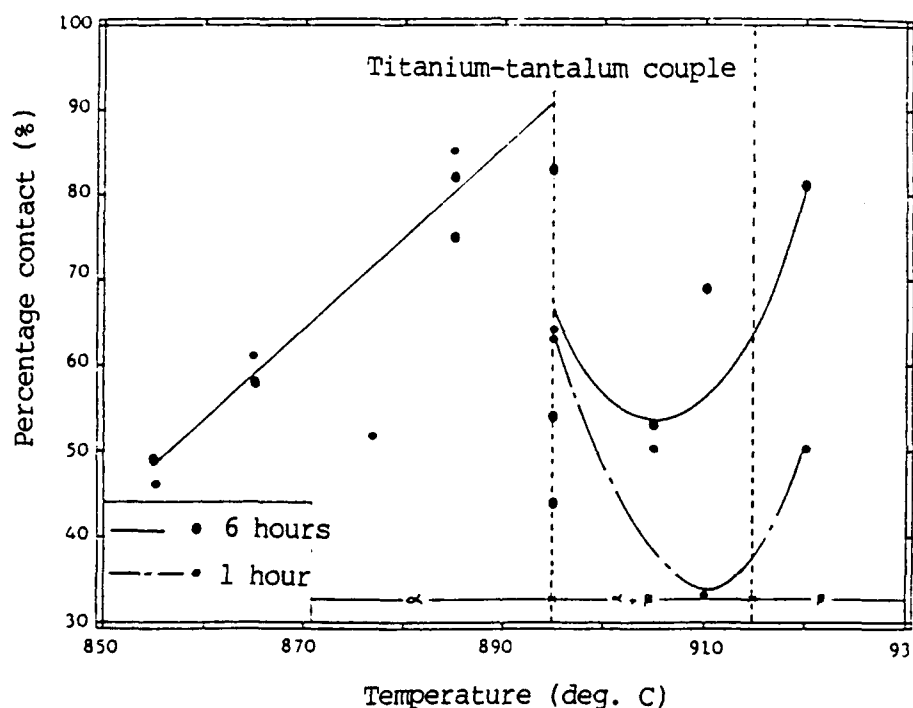


Fig 5 Growth of percentage contact as a function of the bonding temperature of the titanium-tantalum couple (pressure, 2 MPa)

The fact that in the α phase the degree of contact does not increase with bonding period seems to indicate that the resorption of defects by diffusion is very slow, and that it is the first stage of diffusion bonding (the establishment of physical contact by the flow of the rough surface features) which is significant in achieving the union. It would be necessary to speed up the resorption kinetics of the defects by increasing the applied pressure.

These results show that, in order to preserve the benefit in percentage contact obtained during the rise in temperature, it is preferable to operate below the temperature of the start of the $\alpha \rightarrow (\alpha + \beta)$ transformation of titanium.

However, if this temperature is exceeded, the system is still able to repair the damage during the isothermal condition, and even more so as the temperature is raised. However, the test piece will become more deformed, and if the end temperature of the $(\alpha + \beta) \rightarrow \beta$ transformation is exceeded, the titanium will present a large-grained structure.

3.2.1.2 Mechanical properties of the Ti-Ta-Ti assemblies

The variation of the tensile breaking strength of the bonded junctions as a function of the test temperature (Fig 6) is similar to that of the percentage contact (Fig 5), with a consistent growth in the strength of those assemblies whose bonding was carried out in the titanium α phase, and a sharp reduction in breaking strength for tests carried out at a temperature higher than that of the start of the $\alpha \rightarrow (\alpha + \beta)$ transformation. The percentage contact therefore seems to be a good indicator on its own of the quality of the junction. This conclusion may be obvious at first sight; tests carried out on zirconium-tantalum couples¹¹ demonstrate the opposite.

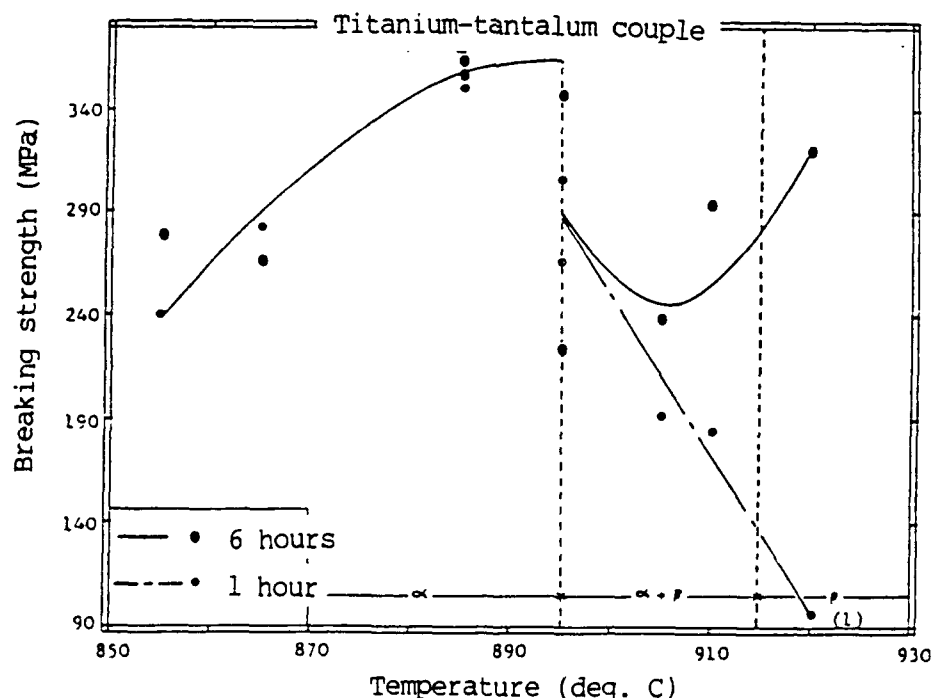


Fig 6 Variation in breaking strength in titanium-tantalum assemblies as a function of bonding temperature (pressure, 2 MPa). (Point 1 does not correlate with percentage contact (Fig 5). The significant density of defects at the junction increases the probability of an early fracture initiated by one of them, made more 'dangerous' by its shape, size or location.)

This investigation shows:

- the percentage contact as defined by us allows the breaking strength of the assemblies to be predicted.
- bonding at temperatures in the ($\alpha + \beta$) and β phases of titanium does not lend itself to the production of good quality junctions, under the conditions of pressure that we applied, and is accompanied by a significant deformation of the test pieces.
- under the conditions of pressure and roughness defined for this investigation, the optimum temperature for diffusion bonding to be found in the α phase, at about 885°C. This allows the achievement of the maximum possible breaking strength (340 to 360 MPa) on account of the configuration of the tensile test sample, as well as a measurable extension at fracture (7% to 14%), while the deformation of the bonded test pieces is limited to acceptably small values.
- under optimum temperature conditions, however, the applied pressure of 2 MPa appears insufficient to achieve assemblies which are strong and ductile enough to cause fracture to occur in the tantalum. It would seem, in fact, that at temperatures in the α phase, the resorption kinetics of defects by diffusion for between 1 h and 6 h may be too slow under the pressure considered (Fig 5). In the remainder of the investigation, the applied pressure was raised to 5 MPa.

3.2.2 Influence of the bonding period

In this investigation we set out to determine the minimum bonding period for the production of strong and ductile assemblies (with fracture occurring in the tantalum), at a pressure of 5 MPa, in the region of the optimum temperature as previously defined (875, 885 and 895°C*), and under the same surface conditions. The maintained temperature periods were 1, 2, 4 and 6 h. The results are given in Table 5.

Measurements of the deformation of the bonded test pieces show that it slows down beyond 4 h of maintained temperature. It is however significant at 895°C. The differences observed in the deformation of the samples bonded in the two series of tests carried out at 885°C are due to the relative positions of the samples in the oven. At the beginning of the process, the samples at the bottom are taken to a higher temperature, and as a consequence are the first to be

* We also operated at 895°C to check our hypotheses.

'crushed' by the action of the applied pressure; their deformation is accompanied by a relaxation effect in the constraint on the sample at the top, which is therefore crushed less. In one series of test carried out at 885°C the samples were at the bottom (causing greater deformation), and in the other, at the top (causing less deformation).

3.2.2.1 Metallographic investigation

The microstructure of the diffusion zone of those junctions bonded between 875 and 895°C resembles that described above (Fig 4) of samples bonded in the α phase. The width of the solid α solution decreases with temperature (reduction in solubility of tantalum in α titanium), while the total width of the diffusion zone increases with temperature and is proportional to the square root of time, in accordance with the laws of diffusion.

With 5 MPa pressure applied during this series of tests, and contrary to earlier observations, the percentage contact of the joints develops with bonding time. Contact between the surfaces is rapidly established during the first hour (with percentage contact between 69% and 80% according to temperature) and more slowly thereafter. The percentage contact exceeds 93% at the end of 4 h of maintained temperature (except at 895°C, where 6 h are required). These results show that, at 5 MPa, the resorption of the residual defects with time proceeds in a measurable manner by a method analogous to sintering; the external pressure accelerates the resorption kinetics. These conclusions are in agreement with the theoretical model of Derby and Wallach¹⁰ relating to homogeneous diffusion bonding.

3.2.2.2 Mechanical properties of the assemblies

The results given in Table 5 show that after one hour of diffusion bonding the maximum strength (350 to 360 MPa) of the assemblies is practically achieved; the breaking strength of the junctions changes little as a function of test duration. It would appear that a percentage contact of about 85% is sufficient to ensure that the assemblies have maximum strength.

As Owczarski *et al*¹² have already shown, the breaking strength is not a characteristic of the quality of the junction, once it has achieved the maximum strength. The extension at fracture and the fracture location* are more sensitive criteria, allowing the classification of junctions having equivalent strengths.

* The 'mixed fractures' (Table 5) relate to sudden fractures initiated at the interface, then absorbed in the tantalum and terminated by plastic flow in the latter; they offer a certain degree of integrity.

Thus we observe that, at temperatures of 875 and 885°C, the extension at fracture increases significantly when the bonding period is increased from 1 to 2 h (Fig 7).

Table 5
Results of bonding tests of the tantalum-titanium
couple as a function of bonding time

Bonding temperature											
	875°C			885°C				895°C			
Period of maintained temperature											
	1 h	2 h	4 h	1 h	2 h	4 h	6 h	1 h	2 h	4 h	6 h
Breaking strength (MPa)	326	342	346	329 342	364 335	368 347	349	335	349	357	351
Extension at fracture (A%)	7	23	46	0 13	37 13	37 42	30	3.5	3	10	39
Fracture* location	Int	Mixed	Ta	Int Int	Mixed Int	Ta Ta	Ta	Int	Int	Int	Ta
Percentage contact (%)	69	85	96	80 79	85.5 83	95 98	94	74	85.5	87	93.5
Deformation of Ti sample (mm)	0.2	0.3	0.5	0.1 0.4	0.3 0.5	0.3 0.7	0.4	0.3	0.5	0.9	0.8
Apparent width of diffusion zone (μm)	12	16	22	10 14	15 20	22 34	25	18	25	36	40
Ti grain size (μm)	168	-	-	183	-	-	212	119	-	-	184

Bonding pressure, 5 MPa; polishing with No.600 paper, * Int, at the interface: Ta, in the tantalum

However, the junction is not yet sufficiently strong after 2 h to withstand without damage the triaxiality of constraints resulting from plastic flow in the tantalum, and fracture occurs at the interface of the two metals. For bonding times of 4 h or greater, the extension at fracture exceeds 30% with these samples, and fracture occurs in the tantalum, indicating that the bonded zone is

sufficiently ductile and strong to withstand the triaxiality of constraints without risk of sudden fracture.

The results of the tests carried out at 895°C, a temperature very close to the $\alpha \rightarrow (\alpha + \beta)$ transformation point, are not so good as the preceding ones; in particular, fracture in the tantalum is obtained only after a bonding period of 6 h. The imminence of structural transformation or a possible temporary increase in temperature beyond the transformation point may explain these results (section 3.2.1).

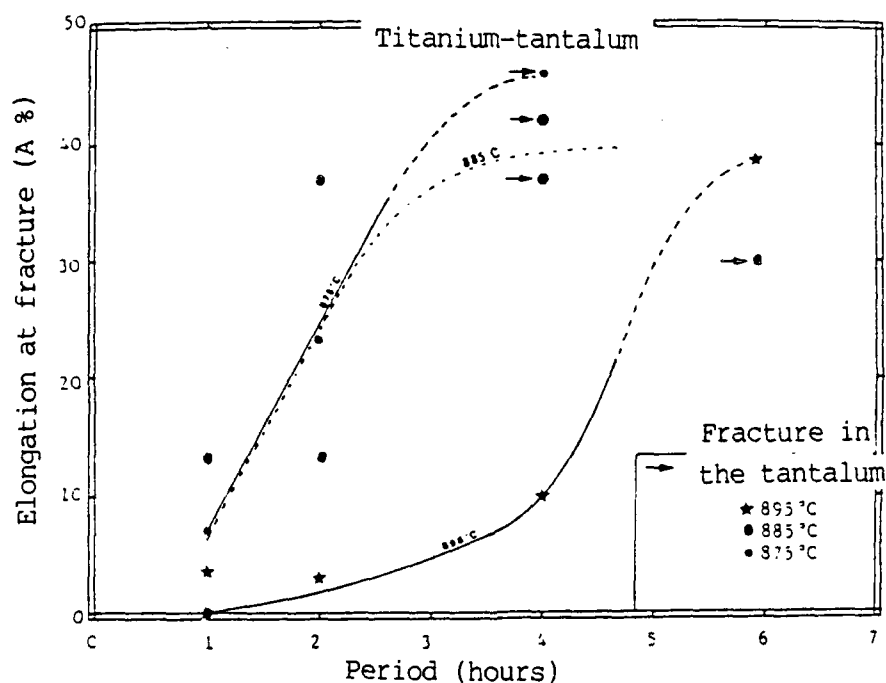


Fig 7 Elongation at fracture of bonded test pieces as a function of the test duration, at different temperatures (pressure, 5 MPa)

It must be noted that fracture of the test pieces in the tantalum is obtained whenever the percentage contact exceeds 93%, and that once again the relationship between this parameter and the mechanical properties of the bonds is confirmed.

This investigation into the period of bonding demonstrates that:

- the period of maintained temperature plays a favourable role in determining the quality of the junction (by improving the percentage contact and consequently the mechanical properties) at the temperatures under

investigation, and at a pressure greater than 2 MPa (equal here to 5 MPa), but is accompanied by deformation of the test pieces, especially at 895°C;

- a pressure of 5 MPa, under the operating conditions defined here, assists in achieving:
- maximum strength (340 to 360 MPa) when the percentage contact reaches 85%;
- fracture of the assemblies occurs in the tantalum after periods of bonding of at least 4 h at temperatures of 875 and 885°C, and at least 6 h at 895°C, corresponding to a percentage contact of at least 93%. These results confirm our previous observations: a temperature of 895°C is unsuitable for bonding, as it is too close to the starting point of structural transformation;
- the extension at fracture and the fracture location are more sensitive criteria than the breaking strength for assessing the quality of a junction; these properties are also directly related to the percentage contact.

3.2.3 Influence of the pressure applied during bonding

These are two effects of pressure: (1) the application of pressure on samples to be diffusion bonded provides support for the samples, encourages the flattening of rough surface features at the start of the test, and speeds the process of residual defect resorption during the period of maintained temperature 5-7; however, at the same time (2) the pressure, applied uniaxially, causes an a priori undesirable deformation of the test pieces.

At this stage the aim of the investigation was therefore to identify the favourable influence of pressure, and limit the negative aspects. In the first place we wished to confirm the conclusion reached by King and Owczarski⁵ relating to homogeneous titanium-titanium diffusion bonding, according to which the removal of pressure was not detrimental if carried out at the end of the first stage, corresponding to the flattening of the rough surface features. Removal of pressure during the test offers the advantage of limiting the deformation of the test pieces and allows information concerning the function of pressure during the diffusion stage to be obtained.

In the second place, in order to limit the deformation of the assemblies, as would be caused by a significant constant uniaxial pressure, we applied an overpressure during the temperature rise.

All these tests were carried out at around the optimum temperature determined above, 875 and 885°C.

3.2.3.1 Tests involving removal of pressure after maintaining temperature for 1 h

We decided to remove the pressure after 1 h of bonding, because the results obtained for this period were already very satisfactory (the strengths of the assemblies being close to the maximum), and the state of progress of the joint seemed to correspond to the end of the first stage as defined by King and Owczarski^{4,5}.

Tables 6 brings together the results of the 1 h trials with those of 4 and 6 h, carried out with and without removal of the 5 MPa pressure. Accordingly, when this was reduced after 1 h of bonding under 4 and 6 h maintained conditions:

- the deformation of the titanium remains at the value obtained for the 1 h tests, in accordance with the required objective;
- the percentage contact, on the other hand, is less than that obtained when the pressure is maintained for the whole of the trial, but greater than that obtained for 1 h bondings; the removal of the pressure causes a slowing of the resorption of the pores, but not a complete cessation;
- the value of the tensile strength of the assemblies is practically the same for all the trials, the percentage contact achieved being sufficient to give the junction maximum strength. However, the extension at fracture is less than that obtained when the pressure is maintained throughout the whole bonding cycle, and greater than that obtained in the 1 h tests, which is in good agreement with the measurements of percentage contact. Fracture of the assemblies in the tantalum is obtained only after 6 h of the maintained condition.

As a consequence, the removal of pressure during the test does not appear to be the most sound solution for limiting the deformation of the test pieces, insofar as pressure plays a significant role in defect resorption kinetics, and its removal must be compensated by an increase in bonding time, which is always penalising.

These results do not completely confirm those of King and Owczarski⁵: the removal of pressure at the end of the first stage (estimated at 1 h in the case of the Ti-Ta assembly) is seen in this investigation to be detrimental to the quality of the junctions. This apparent disagreement is concerned only with the

Table 6

Tests in which the 5 MPa pressure was removed during diffusion bonding of the titanium-tantalum couple

		Bonding temperature							
		875°C				885°C			
		Bonding time							
		1 h	4 h	4 h	6 h	1 h	4 h	4 h	6 h
		Time for which 5 MPa applied							
		heat + 1 h + cool	heat + 4 h + cool	heat + 1 h	heat + 1 h	heat + 1 h + cool	heat + 1 h + cool	heat + 1 h + cool	heat + 1 h
Breaking strength (MPa)		326	346	350	349	342	347	344	352
Extension at fracture (A%)		7	46	17	56	13	42	16	45
Fracture location*		Int	Ta	Int	Ta	Int	Ta	Int	Ta
Percentage contact (%)		69	96	85	93	79	98	79	93
Deformation of Ti sample (mm)		0.2	0.5	0.2	0.2	0.4	0.7	0.3	0.4
Apparent width of diffusion zone (μm)		12	22	24	28	14	34	31	38

*Int, at the interface; Ta, in the tantalum

definition of the stages of diffusion bonding, whose overlapping boundaries cannot be defined precisely. The experimental determination of the end of the first stage, referred to by King and Owczarski, corresponds in actual fact to the 'minimum percentage contact' at which the pressure may be removed with the assurance of a good quality junction, due to the increase in the percentage contact acquired during diffusion. In our case, it would have been necessary to remove the pressure after only the first two hours of bonding (with percentage contact in the region of 85%) and to maintain the temperature for another two hours to achieve 92 to 95% contact.

It may appear surprising to observe an increase in percentage contact after removal of pressure, since for trial carried out at 2 MPa maintained pressure no measurable increase in percentage contact was observed between the 1 h and 6 h tests. This may be explained by the fact that, if the resorption kinetics of the defects depend on the applied pressure, they also depend on the size of the defects, since the sintering pressure (σ_F) grows as the defect size diminishes:

$$\sigma_F = 2\gamma_s/r ,$$

where γ_s is the surface tension of the defect, and r , its radius considered as a sphere, for simplification.

It is likely - and the low measured percentage contact seems to confirm this hypothesis - that at a pressure of 2 MPa applied from the start of the test, the size of the defects, after being subjected to temperature and 1 h of bonding, was too great for their rapid resorption; it would have been necessary to apply an increased pressure (5 MPa, for example) for their resorption. On the other hand, after reaching temperature and holding it for 1 h at 5 MPa, the majority of the defects had probably reached a sufficiently small size for their resorption to be achieved under the influence of the sintering pressure alone, without external pressure.

Continuing these observations, it appeared of interest to examine the influence of the application of a high pressure during the rise in temperature, in order to reduce the number and size of defects present at the start of the maintained temperature, and to try to limit the deformation of the test pieces.

3.2.3.2 Overpressure tests

The procedure consists of:

- application of 50 MPa pressure during heating from ambient temperature to about 750°C, a temperature at which the deformation of titanium accelerates destructively;
- reduction of pressure to 5 MPa, and holding at this value for the remainder of the operation.

Table 7

Results of overpressure tests carried out with the tantalum-titanium couple

	Temperature			
	875°C		885°C	
	Bonding pressure			
	5 MPa	50 MPa →5 MPa	5 MPa	50 MPa →5 MPa
Breaking strength (MPa)	326	351	342	346
Extension at fracture (A%)	7	32	13	42
Location of fracture*				
Location of fracture*	Int	Mixed	Int	Ta
Percentage contact (%)	69	87.5	79	89
Deformation of Ti sample (mm)	0.2	0.3	0.4	0.5
Apparent width of diffusion zone (μm)	12	10	14	16

Polishing with No.600 paper; bonding time, 1 h.

* Int, at interface; Ta, in the tantalum; Mixed, fracture starting at the interface and ending in the tantalum

The first tests were carried out at 875 and 885°C for a bonding time of 1 h (Table 7). Some other results for overpressure tests are presented, together with those relating to the influence of roughness.

Table 7 shows that the application of a temporary overpressure offers advantages in many respects. In fact, it provides:

- limitation of test piece deformation to values approximating to those obtained in tests of the same duration at constant pressure; the overpressure during heating does not induce a net increase in deformation, since the elastic limit of titanium is still sufficiently high at temperatures below 750°C;
- 10 to 20% growth in contact of the junctions and therefore,
- significant improvement in the mechanical properties of the assemblies compared with tests of equivalent duration;
- one quarter of the bonding time necessary to obtain fractures in the tantalum.

The required objective was therefore achieved. The following conclusions may be drawn from this investigation of the influence of applied pressure:

(1) Pressure plays an essentially favourable role in the resorption kinetics of defects, as much during the stage in which rough surfaces are flattened as in the diffusion stage; however, an increase in pressure, if uniaxial, increases the deformation of the test pieces. Furthermore, although the removal of pressure during the test limits the deformation of the test pieces, it is detrimental to the quality of the junction, in so far as the resorption of defects becomes slower. It is therefore necessary to increase the diffusion time in order to obtain assemblies having the equivalent characteristics.

(2) The application of a temporary overpressure is seen to be of advantage with regard to the deformation of the test pieces, the saving in time and the quality of the junctions obtained.

3.3 Optimisation of temperature, pressure and time as a function of roughness

All the preceding tests were carried out on samples polished with No.600 paper, then chemically etched, giving an R_a of around 0.2 μm , which may be obtained industrially by grinding or rolling. It appeared of interest to us to study the influence of greater roughness on the quality of junctions obtained under the optimum operating conditions previously defined, in order to consider other kinds of preparation of industrial surfaces (milling, turning, etc).

Table 8

Results of tests of diffusion bonding of the tantalum-titanium couple as a function of roughness

	Period of maintained temperature										
	4 h 6 h			4 h 6 h			2 h				
	Temperature (°C)										
	Pressure										
	5 MPa 5 MPa 50 MPa →5 MPa			5 MPa 5 MPa 50 MPa →5 MPa			100 MPa (HIP)**				
875 875				885 885			830				
Polishing with paper no.											
600	400	400	400	320	Coarse	600	400	400	400	320	Coarse
346	329	343	324	300		347	349	332	353	347	320
46	7	46	43	6		42	30	9	47	48	6.5
Ta	Int	Ta	Ta	Int		Ta	Ta	Int	Ta	Ta	Int
96	78	97	97	88		98	94	84	97	99	92
0.5	0.5	0.8	0.7	0.8		0.7	0.4	0.7	1	1	1
22	30	32	32	32		34	25	38	40	42	44
-	166	-	-	-		-	212	-	-	-	-

* Int, at the interface: Ta, in the tantalum: ** Hot isostatic pressing

A first test of 6 h at 875 and 885°C and 5 MPa, using no.400 paper for polishing followed by chemical etching ($R_a \approx 0.3 \mu\text{m}$), was immediately seen to be worse than the equivalent tests carried out with no.600 paper (Table 8); contact falls by more than 10% and fracture occurs at the interface. It is clear that a pressure of 5 MPa is insufficient to cause flattening of the rough features from polishing with no.400 paper.

As a consequence, in order to limit the deformation of the test pieces which an increase of pressure would have brought about, cycles of overpressure were applied to very rough samples, *i.e.* roughly machined or polished with no.320 or 400 paper, which were then diffusion bonded at 875 and 885°C for 6 h. The values of R_a corresponding to the above surface conditions are respectively 1 μm , 0.5 μm and 0.3 μm . The results (Table 8) demonstrate that 50 MPa pressure is adequate for flattening during heating the rough features resulting from polishing with no.400 and 320 paper, and allows the production, after the temperature has been maintained for 6 h, of a strong and ductile assembly which breaks in the tantalum with 97% contact. However, for roughly machined samples, the overpressure is seen to be insufficient, and they break at the interface. It is likely that the percentage contact measured in this case is overestimated by reason of the preferential orientation of the machining marks (and therefore of the residual defects) with respect to the plane of examination.

So, as expected, an increase in roughness is detrimental to the quality of the junctions, but it is possible to compensate for this shortcoming by an adequate increase in pressure. If an isostatic press is not available, one may resort to the application of a temporary overpressure, which will limit the deformation of the test pieces and give very satisfactory results. In cases requiring the bonding of test pieces of more complex geometry than those employed in this investigation, in particular those having symmetry of revolution, a test with comparable overpressure may be achieved using the 'expanding insert technique'. This consists of exploiting the difference between the expansion coefficients (those of the test pieces being bonded, or of a foreign insert and the test pieces) to apply the overpressure; also of using the relaxation phenomena of the metal at temperature in order to reduce the pressure over a period of time.

We were offered the opportunity of carrying out a test using the isostatic press at CEA. The available pressure (100 MPa) being very much greater than that with which we were working, we reduced the temperature of the test. The bonding was therefore carried out at 830°C, at a constant pressure of 100 MPa maintained over the two-hour bonding period, and was performed simultaneously with samples

which were either roughly machined or polished with no.600, 400 or 320 paper. All the test samples obtained fractured in the tantalum, and none of the junctions exhibited residual defects at the interface, whatever the initial state of the surface. Diffusion bonding performed in the isostatic press, although expensive, offers many advantages both in respect of test pieces deformation and the ability to vary the operating parameters. The high pressures employed in this technique allow considerable reductions in temperature and/or increase in roughness of the surfaces to be bonded.

4 CONCLUSIONS

- (1) The production of titanium-tantalum assemblies stronger than tantalum ($R_m = 250$ MPa) is quite possible for a number of combinations of the operating parameters. The tensile strength of diffusion-welded junctions may be greater than 340 to 360 MPa in the best cases, and fracture then occurs in the tantalum, as evidenced by the tensile test pieces.
- (2) The Kirkendall effect observed in the first part of this investigation does not appear detrimental to the production by diffusion of titanium-tantalum junctions, for bonding times of between 1 and 6 h.
- (3) The percentage contact, as we have defined it, provides a sufficiently accurate indication of the characteristics and mechanical properties of the assemblies; those junctions whose percentage contact reaches 85% demonstrate a tensile strength practically equal to the maximum (340 to 360 MPa); a percentage contact of around 93% causes fracture to occur in the tantalum.
- (4) The optimum bonding temperature at low pressure (2 or 5 MPa) ranges from 875 to 885°C in the α phase of the titanium. It is not advisable to bond at low pressure in the $(\alpha + \beta)$ and β phases of titanium (at temperatures of at least 895°C), since passing beyond the $\alpha \rightarrow (\alpha + \beta)$ transformation temperature is accompanied by damage to the junctions and significant deformation of the test pieces. However, bonding at high pressure in the $(\alpha + \beta)$ phase (in an isostatic press, to avoid deformation) should be possible, with the effect of the pressure compensating that of the structural transformation.
- (5) If the pressure is sufficiently high, maintaining the temperature for an extended time has a beneficial effect on the quality of the junction, but is accompanied by deformation of the test pieces. The optimum bonding time is a function of other experimental parameters.
- (6) Pressure plays a decisive role in the resorption kinetics of defects. This is as significant during the stage of flattening the rough surface features while

heating as during diffusion, when the sintering pressure is added to the applied pressure. A pressure of 2 MPa is insufficient to obtain around 93% contact in a reasonable time (less than or equal to 6 h), but it may be obtained at a pressure of 5 MPa.

However, in the case of uniaxial pressure, an increase causes a more significant deformation of the test pieces. The removal of pressure during the test is detrimental to the quality of the junctions because if the test pieces are deformed less, the resorption kinetics of the defects slow down, and it then becomes necessary to prolong the bonding time in order to obtain assemblies of equivalent quality. On the other hand, the application of a temporary overpressure is seen to be of advantage with respect to deformation of test pieces, saving of time and the quality of the junctions obtained.

(7) An increase in roughness of the surfaces to be assembled is detrimental to the ductility of the assembly, even under the previously determined optimum conditions. However, it is always possible to compensate for this by the application of a temporary overpressure, or by a large enough increase in the permanent pressure (which implies operation under isostatic conditions if deformation of the test pieces is to be avoided).

This investigation proves that it is possible to find, within technical and other constraints, a combination of the four principal parameters which will allow the diffusion bonding of tantalum and titanium to be carried out successfully. The limits of each of the operating parameters are specified in Table 9 (for the case of pure tantalum and commercially pure titanium). All these results appear to be readily transferable, *mutatis mutandis*, to examples of diffusion bonding of alloy of titanium and tantalum. We have in fact successfully bonded TA6V and tantalum at 915 to 925°C and 5 MPa, over a period of 45 minutes: the assemblies fractured in the tantalum at 380 MPa.

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Table 9

Temperature	
Optimum value at low pressure (2 to 5 MPa)	875 to 885°C
Lower limit at high pressure (100 MPa)	< 830°C
Upper limit at low pressure (2 to 5 MPa)	895°C (structural transformation of the titanium employed).
Upper limit at pressure higher than 5 MPa	No limit, but from 915°C deformation of the test pieces (except under isostatic conditions) and considerable increase in grain size.
Pressure	
Lower limit	Between 2 MPa (exclusive) and 5 MPa (inclusive).
Upper limit	According to the permissible deformation, also dependent on the temperature (<i>eg</i> 10 MPa at 865°C for a deformation of the order of 10%).
With isostatic pressure	
Lower limit	As for uniaxial.
Upper limit	Press capacity.
Duration of the maintained condition	Function of temperature and pressure, <i>eg</i> 4 h minimum at the optimum temperature and 5 MPa. 1 h minimum at the optimum temperature and with a temporary overpressure (50 MPa reducing to 5 MPa, from 750°C.
Roughness	Function of the preceding parameters, especially temperature and pressure, <i>eg</i> maximum R_a of about 1 μm with temporary overpressure.

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